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Characterisation of an automated Dual Piston Pressure Swing Adsorption (DP-PSA) system

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Abstract

The drive to fast Pressure Swing Adsorption (PSA) processes leads to the challenge of designing and analysing experiments that allow the testing of adsorbents under fast cycle conditions. Here we present the Dual Piston-Pressure Swing Adsorption (DP-PSA) system and the accompanying mathematical model designed for this purpose. The comparisons of experimental and simulation results show that for slow cycles often an isothermal model is sufficient. However, for fast cycles the temperature profile over the cycle has to be taken into account to describe accurately the experimental curves; this is essential for the estimation of the parameters of the adsorbent material under fast cycle conditions.

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Keywords: Pressure swing adsorption; piston driven PSA; fast cycles; material screening; mathematical model; non-isothermal;

1. Introduction

Adsorption processes such as Pressure Swing Adsorption (PSA) and Vacuum Swing Adsorption (VSA) are receiving considerable interest for Carbon Capture and Storage applications [1]. This is due to the potential high productivity of adsorption processes [2]. To achieve high productivity cycle times have to decrease from a few minutes to a few seconds. This leads to the challenge of designing experiments that allow testing materials and process configurations under fast cycle conditions, in order to be able to determine the physical parameters that allow reliable predictions of process performance.

PSA technology was introduced by Skarstrom [3] for air drying using a simple dual-bed unit. Since then the productivity and efficiency of PSA processes has been improved by increasing the number of adsorption beds and reducing the cycle time [4]. Especially the Rapid PSA (RPSA) process which was

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introduced in the early 1970s [5] has the potential to utilise efficiently the adsorbent material and thus to achieve high productivity. A variant of the RPSA process is a piston driven PSA process which was introduced by Eriksson [6] and extended to the Dual Piston PSA (DP-PSA) process by Keller and Kuo [7]. Here the movement of the pistons induces cyclic gas flow and pressure variations along the column; this parametric pumping can lead to a highly productive separation processes [4]. To design a RPSA system with high productivity it is essential to establish the adsorbent characteristics at these fast cycle configurations. While the DP-PSA process can reach high productivities it is also particularly well suited for the testing of adsorbent materials under fast cycle configurations. By changing the stroke lengths, phase angles and cycle times of the two pistons a large variety of different experiments can be performed; thus testing the adsorbents under different conditions. To take full advantage of the DP-PSA system a mathematical model which accurately describes the system is required [8-10].

Here we present a semi-automatic DP-PSA system for the characterisation of adsorbents. We describe the experimental configuration with particular emphasis on the independent and automatic control of the two pistons. A non-isothermal mathematical model of the DP-PSA system is developed and compared to experimental data.

Nomenclature

c_i	concentration of component i , mol m^{-3}
d_p	adsorbent pellet diameter, m
F	flow rate, mol s^{-1}
k_i	LDF parameter of component i , s^{-1}
P	pressure, Pa
q_i	adsorbed concentration of component i , mol m^{-3}
t	time, s
t_c	cycle time, s
T_f	fluid temperature, K
T_p	pellet temperature, K
T_w	wall temperature, K
U_f	internal energy in the gas phase, J m^{-3}
U_p	internal energy in the adsorbent pellet, J m^{-3}
v	interstitial velocity, m s^{-1}
V	piston volume, m^3
V_c	column volume, m^3
x_i	mole fraction of component i
ε	bed void fraction
μ	viscosity, Pa s
ρ_f	fluid density, kg m^{-3}

2. Materials and methods

2.1. Experimental setup and control instrumentation

A schematic of the DP-PSA system is shown in Fig. 1. Briefly, the DP-PSA system consists of an adsorption column which is connected at the top and bottom to two pistons, which are controlled independently. The complete system is enclosed in an oven to control the surrounding temperature. The two pistons are driven by an ABB drive and independently controlled by a real-time computer, National Instruments CompactRIO 9022, with a Labview interface; allowing accurate and fast piston control down to 1 second cycles. The automated and independent control of the two pistons facilitates the running of adsorption experiments with widely differing pressure and flow profiles: compression ratios between 1 and 10 and flow rates ranging from ml/s to l/s. The real-time computer also handles the data acquisition from two pressure transducers and five thermocouples. The pressure transducers are placed at the top and bottom of the column to monitor the absolute pressure in the system and the pressure drop across the column. Four of the thermocouples are placed inside the column: two in the gas phase and two inside 13X pellets. This allows the monitoring of the temperature profile in both the gas and solid phase. The fifth thermocouple is placed just outside the column to monitor the oven temperature. After manually filling the system with a gas mixture and setting the oven temperature (20°C to 200°C) a series of experiments can be run automatically by the real-time computer.

Several interface modules are connected to the CompactRIO for the different control and monitoring tasks. The general configuration of the ABB drives, such as acceleration and deceleration, is transmitted by serial communication while the actual position reference is transmitted by an analog signal. This setup reduces the data transmission time and is crucial for the accurate control of fast cycles. The pressure signals are recorded through a serial connection and the thermocouple signals through a dedicated thermocouple input module. A user interface was built on the Labview front panel. This interface is run on the host computer before the code is deployed to the CompactRIO. In this interface the user can define a series of experiments through the piston parameters: cycle time, stroke and offset. Once the series of experiments is defined the Labview code is deployed to the CompactRIO which then runs the code independently of the host computer without further user input. During the experiments the temperature and pressure profiles are stored on the CompactRIO and only transferred to the host computer once the series of experiments is completed.

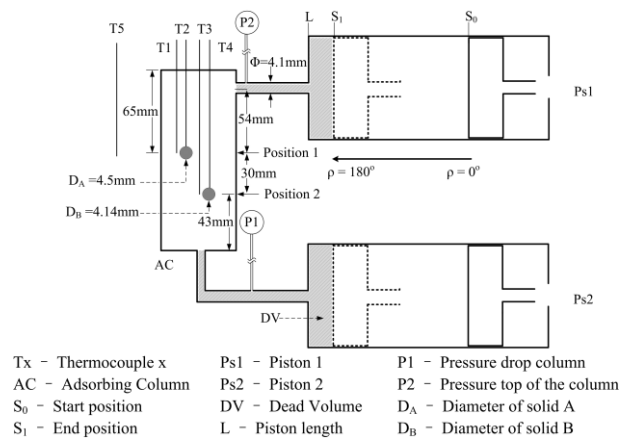


Fig. 1. Schematic showing the DP-PSA system

2.2. Experimental procedure

Before each new series of experiments, the adsorbent material inside the column has to be regenerated so that adsorbed gas and water molecules are desorbed. The oven is set to 200 °C and the column is connected to an oil-free vacuum pump for at least 12 hours. After the regeneration the oven is set to the experimental temperature and the system is left to cool down until it has reached the desired temperature. Before feeding the gas to the system, the two pistons are moved in a slow, out-of-phase configuration so that the total volume remains constant. This brings the seals in the piston to the working temperature. The gas for the experiment is prepared in cylinders which are also located in the oven. Gas is fed in to the column by quickly opening and closing the valve between the column and the dosing system. By comparing the pressure of the dosing system, which has a known volume, before and after the valve operation, the amount of gas dosed into the column can be calculated. Once the system temperature and pressure have stabilised the series of experiments is started.

3. Mathematical model

The mathematical model describing the DP-PSA system is a non-isothermal extension of the model described by Farooq's and Ruthven's groups [8, 9]. The model is based on the following assumptions:

- Axial dispersed plug flow
- Pressure drop described by the Ergun equation
- Non-isothermal operation
- Linear Driving Force (LDF) mass transfer model
- Langmuir adsorption isotherm
- Flow resistance between the pistons and the column
- Pistons are perfectly mixed, i.e. concentrations and temperature are uniform
- The system dead volume is at the top of the piston chamber

3.1. Governing equations for the column

The material balance along the column for axial dispersed plug flow is given by

$$\frac{\partial c_i}{\partial t} + \frac{(1-\varepsilon)}{\varepsilon} \cdot \frac{\partial q_i}{\partial t} + \frac{\partial(c_i \cdot v)}{\partial z} + \frac{\partial J_i}{\partial z} = 0$$

Here i goes from 1 to the number of components N_c . The mass transfer in the adsorbent is given by

$$\frac{dq_i}{dt} = k_i (q_i^* - q_i)$$

where q_i^* is the sorbate concentration at equilibrium. This value depends on the multi-component Langmuir isotherm

$$q_i^* = \frac{q_{i,s} b_{i,0} \exp\left(\frac{-\Delta\tilde{H}_i}{RT}\right) P x_i}{1 + \sum_{j=1}^{N_c} b_{j,0} \exp\left(\frac{-\Delta\tilde{H}_j}{RT}\right) P x_j}$$

The energy balance in the column is written in terms of the internal energy in the fluid and solid phase

$$\varepsilon \frac{\partial U_f}{\partial t} + (1 - \varepsilon) \frac{\partial U_p}{\partial t} + \varepsilon \frac{\partial (H_f \cdot u)}{\partial z} + \frac{\partial J_T}{\partial z} + \sum_{i=1}^{N_c} \frac{\partial (J_i \tilde{H}_i)}{\partial z} + h_w \frac{A_c}{V_c} (T_f - T_w) = 0$$

$$\frac{dU_p}{dt} = h \frac{A_p}{V_p} (T_f - T_p)$$

The pressure in the system is calculated from the gas phase concentration and temperature through the ideal gas law. The interstitial flow velocity v is calculated from the Ergun equation

$$-\frac{\partial P}{\partial z} = \frac{150\mu(1-\varepsilon)^2}{d_p^2 \varepsilon^2} v + \frac{1.75\rho_f(1-\varepsilon)}{d_p \varepsilon} v|v|$$

The boundary equations for the mass and energy balance in the column are given by the Danckwert's boundary conditions which can be written concisely as

$$J_T|_{z=0} = \frac{v+|v|}{2} (H_{f,0-} - H_{f,0}), \quad J_T|_{z=L_c} = \frac{v-|v|}{2} (H_{f,L_c+} - H_{f,L_c})$$

$$J_i|_{z=0} = \frac{v+|v|}{2} (c_{i,0-} - c_{i,0}), \quad J_i|_{z=L_c} = \frac{v-|v|}{2} (c_{i,L_c+} - c_{i,L_c})$$

The velocities at the column boundaries are calculated from the resistance term between the pistons and the column. Initially the column is at uniform pressure, temperature and gas phase concentration. The adsorbent material is assumed to be in equilibrium with the set gas phase concentration.

3.2. Governing equations for the pistons

The piston position can be described by the following equation

$$S(t) = S_0 + \frac{S_1 - S_0}{2} (1 - \cos(\omega t + \varphi))$$

where S_0 and S_1 are the start and end position of the piston, respectively, $\omega = 2\pi/t_c$ is the cycle frequency and φ the initial piston offset. The material balance in the pistons is given by

$$\frac{d(c_i V)}{dt} = \frac{F + |F|}{2} \frac{c_{i,n}}{c_{T,n}} + \frac{F - |F|}{2} \frac{c_i}{c_T}$$

where the subscript n indicates the concentration in the neighbouring unit, i.e. the concentration at the top or bottom of the column depending on the piston. The flow rate at the piston outlet is modelled through a resistance term

$$F = \kappa(P_n - P).$$

The energy balance is written in terms of internal energy

$$\frac{1}{V} \frac{dU_f}{dt} = \frac{F\tilde{H}}{V} - \frac{P}{V} \frac{dV}{dt} - h_w \frac{A_c}{V} (T_f - T_w)$$

3.3. Numerical simulation of the governing equations

The governing partial differential algebraic equations are solved with the Method of Lines. Here the spatial dimension is discretised in space using a flux-limited finite volume method and the resulting set of ordinary differential algebraic equations is integrated in time with the variable time step, variable order backward differentiation formulas implemented in SUNDIALS [11].

4. Experimental runs

Experiments with pure helium and mixtures of nitrogen and carbon dioxide were performed with an empty column and with a column packed with zeolite 13X pellets. This hierarchy of experiments is used for the sequential characterisation and validation of the experimental system and mathematical model.

4.1. Helium runs

The runs with helium, which is assumed to be non-adsorbing, in both the packed and empty column allow the characterisation of the dead volumes and the matching of experiments and simulations under non-adsorbing conditions. The dead volumes for the empty and packed column are calculated from stopped flow experiments: the piston is moved in several steps from position S_0 to position S_1 (see Fig. 1). From the pressure and position data the dead volumes are calculated from the ideal gas law.

Fig. 2 (a) shows that the simulated pressure profile of the Helium run with $t_c=10s$ for the isothermal and non-isothermal model agrees well with the experiment data. This confirms that the dead volumes are close to the actual values. Furthermore, the overlap of the isothermal and non-isothermal simulation curves suggests that for slow cycles an isothermal model which is computationally less expensive and requires fewer physical parameters is sufficient. Fig. 2 (b) shows the experimental and simulated pressure drop for a fast Helium run. While the simulated pressure drop profile is not exactly the same as the experimental profile, the qualitative shape of the profile and the magnitude of the pressure drop are well described by the Ergun equation.

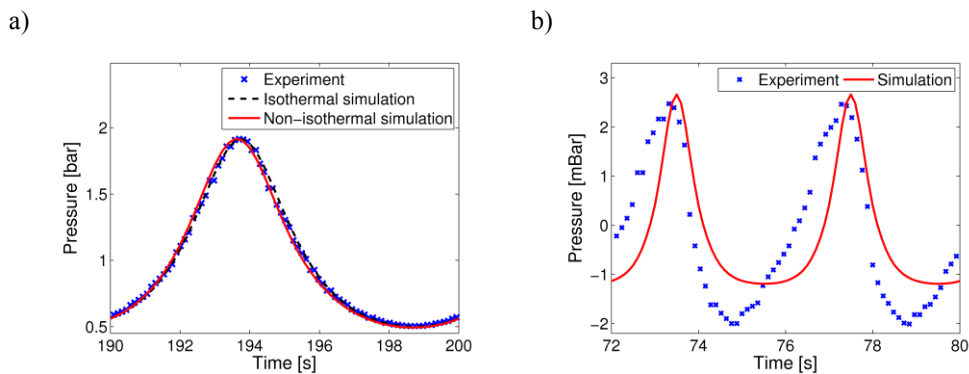


Fig. 2. (a) Pressure profile for the Helium run with $t_c=10s$ and (b) pressure drop across the column for the Helium run with $t_c=4s$.

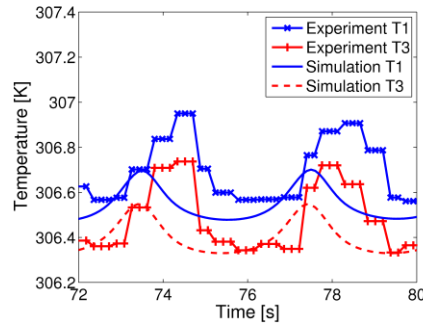


Fig. 3. Experimental and simulated temperature profiles for the two thermocouples in the gas phase. See Fig. 1 for the location of the thermocouples T1 and T3.

Fig. 3 shows the experimental and simulated temperature profile of the two thermocouples inside the gas phase in the column for a Helium run with $t_c=4s$ and a phase angle of 0.5π between the two pistons, i.e. piston Ps1 is a quarter through its cycle from S_0 to S_1 and back when piston Ps2 starts its cycle. While the simulated temperature profile is shifted from the experimental profile, it clearly confirms the temperature gradient along the column seen in the experimental data. This temperature gradient is generated by the asymmetric pressure profile (see Fig.2 (a)): the two pistons perform the compression and expansion phases of the cycle with different pressures and thus require different amounts of work. Piston Ps2 is compressing the gas at high pressures and expanding at low pressures while piston Ps1 is compressing at low pressures and expanding at high pressures. Thus the work required at piston Ps2 is larger than the work required at piston Ps1. This in turn leads to a higher temperature in piston Ps2 and to a temperature gradient along the column as shown in Fig. 3.

4.2. CO_2/N_2 runs

The helium runs are followed by experiments with a 50:50 mixture of CO_2 and N_2 at various cycle times and piston offsets. The adsorption isotherms for CO_2 and N_2 are taken from the literature [12]. The kinetics are under macropore diffusion control [8] and the LDF parameters are calculated from the effective pore diffusivity [2].

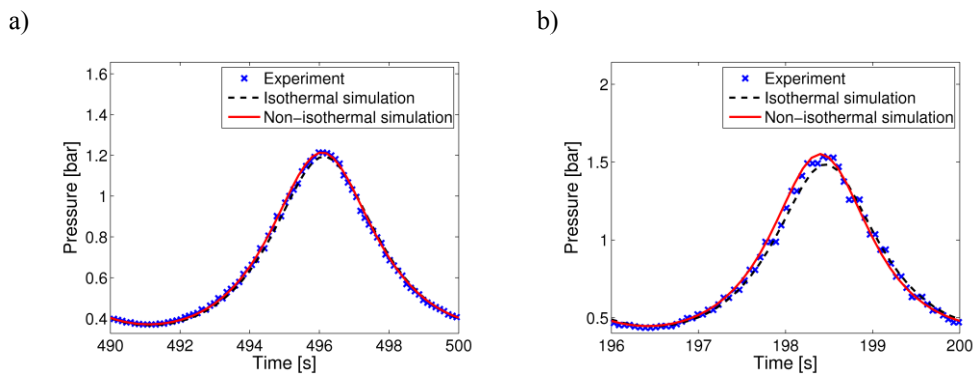


Fig. 4. Pressure profile for the mixture run with (a) $t_c=10s$ and (b) $t_c=4s$.

As suggested by the helium experiments the isothermal model showed good agreement with the experimental data for cycle times larger than 10s; see Fig. 4 (a). However, for fast cycles the isothermal model starts to deviate from the experimental data. This is particularly evident at the pressure peak in Fig. 4 (b). Here the heat generated by the adsorption lowers the isotherm so that less CO₂ is adsorbed; thus more molecules remain in the gas phase and in turn the pressure is higher than predicted by the isothermal model. The temperature also affects the mass transfer kinetics but this relationship is not yet included in the mathematical model. This discrepancy between the isothermal model and the experimental data will increase with decreasing cycle times. On the other hand, the non-isothermal model developed in section 3 matches the experimental pressure profile almost perfectly.

5. Conclusion

Here we have presented an automated DP-PSA system for the characterisation of novel adsorbents. The instrumentation of the DP-PSA system and the non-isothermal model for the analysis of the experimental data was described and shown to describe accurately single component systems. We performed experiments with inert and adsorbing gases to characterise the system and to validate the mathematical model. These experiments revealed that the isothermal model is usually sufficient for slow cycles, typically for cycle times greater than 10 s and pressure ratios of 2-3. However, we showed that for fast cycles the temperature profiles in the system have to be considered since the temperature influences the equilibrium and kinetic parameters of the adsorbents.

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References

- [1] Liu Z, Grande CA, Li P, Yu J, Rodrigues AE. Adsorption and Desorption of Carbon Dioxide and Nitrogen on Zeolite 5A. *Separ Sci Technol* 2011;**46**:434–451.
- [2] Ruthven DM. *Principles of adsorption and adsorption processes*. New York: Wiley-Interscience; 1984.
- [3] Skarstrom CW. US Patent 3,237,377. 1966.
- [4] Ruthven DM, Farooq S, Knabel KS. *Pressure Swing Adsorption*. New York: VCH; 1994.
- [5] Turnock PH, Kadlec RH. Separation of Nitrogen and Methane via Periodic Adsorption. *AIChE J* 1971;**17**(2):335–342.
- [6] Eriksson R. US Patent 4,169,715. 1979.
- [7] Keller GE, Kuo CHA. US Patent 4,354,859. 1982.
- [8] Arvind R, Farooq S, Ruthven DM. Analysis of a piston PSA process for air separation. *Chem Eng Sci* 2002;**57**:419–433.
- [9] Farooq S, Thaeon C, Ruthven DM. Numerical simulation of a parallel-passage piston-driven PSA unit. *Sep Purif Technol* 1998;**13**:181–193.
- [10] Singh K, Jones J. Numerical simulation of air separation by piston-driven pressure swing adsorption. *Chem Eng Sci* 1997;**52**(18):3133–3145.
- [11] Hindmarsh AC, Brown PN, Grant KE, Lee SL, Serban R, Shumaker DE, Woodward CS. Sundials: Suite of Nonlinear and Differential/Algebraic Equation Solvers. *ACM T Math Software* 2005;**31**:363–396.
- [12] Xiao P, Zhang J, Webley PA, Li G, Singh R, Todd RS. Capture of CO₂ from flue gas streams with zeolite 13X by vacuum-pressure swing adsorption. *Adsorption* 2008;**14**(4-5):575–582.